ARO 27321.1-EL-S&I

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"Thin Crystalline InP on Insulating Substrates"

FINAL TECHNICAL REPORT
(CLIN 0002AB)

20030210044

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Period of Effort:
August 15, 1989 to February 15, 1990

U.S Army Research Office
Contract No. DAAL03-89-C-0025

Approved for Public Release;
Distribution Unlimited

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION	1b. RESTRICTIVE MARKINGS
Unclassified	

2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION / AVAILABILITY OF REPORT
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE	Approved for Public Release; Distribution Unlimited

4. PERFORMING ORGANIZATION REPORT NUMBER(S)	5. MONITORING ORGANIZATION REPORT NUMBER(S)
	ARO 27321.1-EL-SBI

6a. NAME OF PERFORMING ORGANIZATION	6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION
AstroPower, Inc.		U. S. Army Research Office

6c. ADDRESS (City, State, and ZIP Code)	7b. ADDRESS (City, State, and ZIP Code)
30 Lovett Avenue Newark, DE 19711	P. O. Box 12211 Research Triangle Park, NC 27709-2211

8a. NAME OF FUNDING / SPONSORING ORGANIZATION	8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
U. S. Army Research Office		Contract No. DAAL03-89-C-0025

8c. ADDRESS (City, State, and ZIP Code)	10. SOURCE OF FUNDING NUMBERS			
	PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO.
P. O. Box 12211 Research Triangle Park, NC 27709-2211				

11. TITLE (Include Security Classification)

"Thin Crystalline InP on Insulating Substrates"

12. PERSONAL AUTHOR(S) Collins, Sandra R.; McNeely, James B.; Barnett, Allen M.

13a. TYPE OF REPORT Final	13b. TIME COVERED FROM 8-15-89 TO 2-15-90	14. DATE OF REPORT (Year, Month, Day) 1990 May 14	15. PAGE COUNT

16. SUPPLEMENTARY NOTATION The view, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

17.	COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB-GROUP	InP, Thin-Film, Radiation-Hard, Large Area, Integrated Optoelectronics

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

This program has successfully developed device quality InP films on insulating substrates. A low cost deposition technique was established which produced mechanically stable, stoichiometric InP films on insulating substrates. The substrate has a thermal expansion coefficient matched to InP for temperatures up to 600°C. Device quality was established by the fabrication and successful operation of Schottky barrier diodes. Device performance comparisons of these devices with identical structures on large grain commercial polycrystalline InP material and LEC single crystal InP material provided a relative basis for analysis of material quality. Device performance was found to be dependent on the grain size of the InP films.

The quality of the InP/Glass photodiodes fabricated and tested during this program indicate the high device potential of these films for grain sizes exceeding 10 microns. Larger grain InP films are needed to provide more conclusive findings on the optimum grain size for device applications. (JS)

20. DISTRIBUTION / AVAILABILITY OF ABSTRACT	21. ABSTRACT SECURITY CLASSIFICATION
☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT ☐ DTIC USERS	Unclassified

22a. NAME OF RESPONSIBLE INDIVIDUAL	22b. TELEPHONE (Include Area Code)	22c. OFFICE SYMBOL
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DD FORM 1473, 84 MAR 83 APR edition may be used until exhausted. All other editions are obsolete

1.0 SUMMARY

This program has successfully developed device quality InP films on insulating substrates. A low cost deposition technique was established which produced mechanically stable, stoichiometric InP films on insulating substrates. The substrate has a thermal expansion coefficient matched to InP for temperatures up to 600°C. The device quality of the films was verified by the fabrication and successful operation of Schottky barrier diodes. Device performance comparisons of these devices with identical structures on large grain commercial polycrystalline InP material and LEC single crystal InP material provided a relative basis for material quality. Device performance was found to be dependent on grain size.

2.0 PURPOSE OF WORK

The goal of this Phase I program was to demonstrate device-quality, large-grain, thin films of InP on insulating substrates. The following list of tasks outline the main objectives of this program as stated in the Phase I proposal.

1) Design and select optimal insulating substrate for the thin-film crystalline InP.

2) Establish a technique for depositing uniform InP thin films on insulating substrates.

3) Apply zone melt recrystallization (ZMR) technology to the InP thin films. Determine encapsulant to maintain InP stoichiometry.

4) Characterize thin-film crystalline InP layers on the insulating substrates.

5) Fabricate and test definitive basic device configurations indicative of integrated optic functionality, such as a light-emitting diode and/or a field-effect transistor on the InP/substrate.
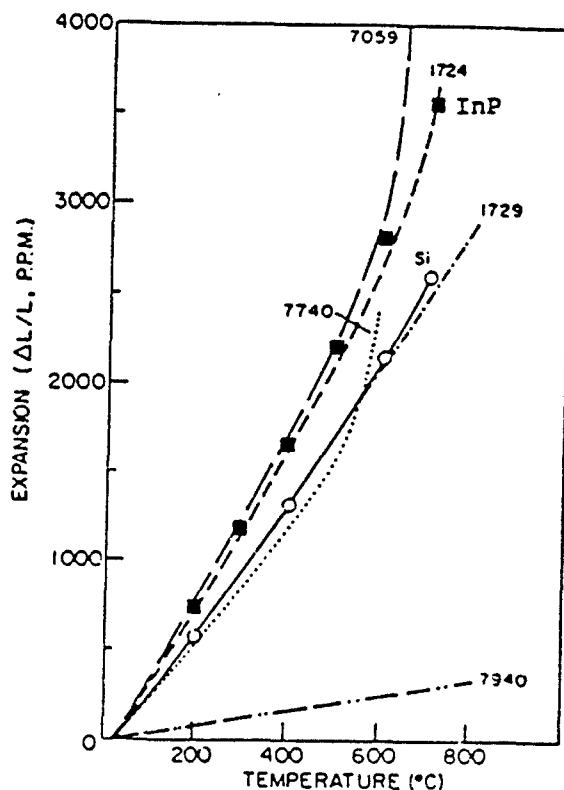
3.0 WORK CARRIED OUT

3.1 Selection of the Insulating Substrate

The selection of the candidate substrate materials for the deposition of the InP films was based on an evaluation of the substrate's chemical, electrical, mechanical, and structural properties, and the behavior of these properties as a function of the temperatures encountered during film deposition and processing. Chemically, the composition of the substrate material was evaluated

to ensure that impurities would not be introduced into the active InP film which would deleteriously affect the operation of the subsequently fabricated devices. Electrically, the substrate was selected to possess an insulating interface with the InP film. Mechanically, it was required that the InP/substrate structure contain a minimum of built-in stress to minimize the production of stress relieving lattice defects which could degrade device performance.

A major contribution to the built-in stress for any layered structure is the mismatch in thermal expansion coefficient existing between the two layers comprising the structure. This mismatch causes the layer with the lower expansion coefficient to be in compression when the temperature of the structure is lowered from the InP film deposition temperature. Conversely, the layer with the higher thermal expansion coefficient is under tension under these circumstances. Therefore, it is advantageous to thermally match the substrate by selecting a material with a thermal expansion coefficient very close to that of InP over the temperature range of interest.

Figure 1. Linear Expansion Coefficient as a Function of Temperature for Several Commercial Glass Substrates, InP, and Silicon.

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It was decided that glass is the most attractive substrate material. Glass is inexpensive, many varieties in large sheet form are available, it is transparent across the solar spectrum, and it is smooth. Due to the amorphous nature of glass, it is not possible to achieve lattice matching, however, the smooth surface presents a passive surface which allows arriving atoms to migrate and eventually congregate at random nucleation sites, thus producing a polycrystalline film. Figure 1 shows the relative expansion as a function of temperature of several candidate glasses (identified by their Corning Glass Works code numbers) as well as InP and silicon.

Three types of glass substrates were investigated during this program. Background information pertaining to these three insulating materials is provided below:

Corning Glass 7059: 7059 provides good thermal expansion coefficient matching to InP from 23°C up to 600°C. It is available in drawn sheets and is ready for InP deposition directly from the manufacturer. This particular glass is used as an optical waveguide material [1].

Corning Glass 1724: This glass provides a close match in thermal expansion coefficient to InP from 23°C up to and beyond 700°C. It is available in powder form and requires processing to obtain necessary substrate structure. This powder, to date, has formed poor quality substrates (brittle and nonuniform). As an alternative, silicon wafers (test wafer grade) were coated with a film of 1724 glass.

GE Glass 177: The thermal expansion coefficient of 177 is closely matched to silicon. Test grade silicon wafers were also coated with this glass to provide a third substrate choice.

All three glass substrate candidates were tested as potential substrates for deposition of InP films. The substrates were subjected to temperatures representative of the deposition and recrystallization process. The 1724 and 177 glass layers cracked, bubbled, and deformed, failing to provide the required mechanical and structural properties required of a desirable InP/Insulator structure.

The 7059 glass substrates have been used throughout InP film development. These substrates did not deform at temperatures required for InP film deposition and processing. 7059 glass is a barium borosilicate compound that is not expected to introduce any impurities into the InP film which would degrade device performance. Accordingly, 7059 glass substrates have been considered the glass substrate of choice for this program.

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3.2 Grain Size of InP Films on Insulating Substrates

The 7059 glass substrate surface is smooth and presents a passive surface which allows arriving atoms to migrate and eventually congregate at random nucleation sites. This yields polycrystalline material with relatively small grain dimensions. It was found that the grain size of the as-deposited InP films increased with increasing deposition temperature. The temperature dependence of grain size is illustrated by the series of photomicrographs and corresponding deposition temperatures given in Figure 3 on the next page. Note that deposition time was held constant for each of the films.

The grain size of the as-deposited InP films has been limited to about 10 micron dimensions on untextured glass. Three methods were used to increase grain size beyond the as-deposited dimensions: i) texturing of the glass surface, ii) post-deposition annealing, and iii) recrystallization using ZMR techniques.

Pre-deposition surface texturing of the glass substrate was achieved by using sandblasting. The grain size of the as-deposited films did not appear to increase with substrate surface texturing, but the film surface did become less smooth. From grain to grain, the surface height changed as much as 3 to 8 microns which would prove problematic for subsequent film processing steps such as photolithography. Figure 2 below shows a photomicrograph of the surface morphology of an InP film deposited on a 7059 glass substrate with a sandblasted surface.

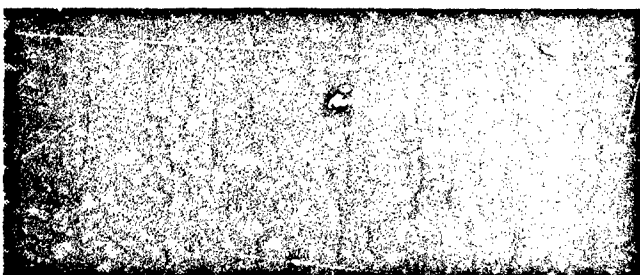(A) Original Sandblasted
    Glass Surface
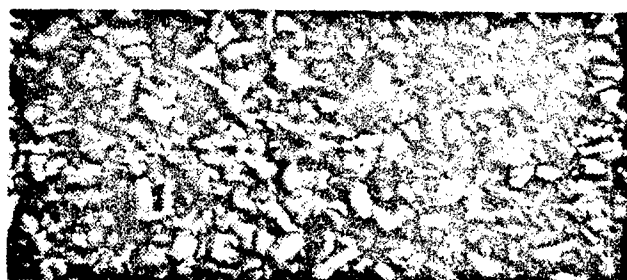
(B) As-Deposited Film
    Morphology

Figure 2. Surface Morphology of InP Film Deposited on a Roughened Glass Surface (Marker = 10 µm):
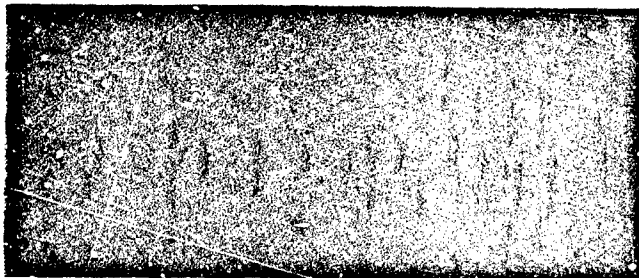
4

A) Sample #7171
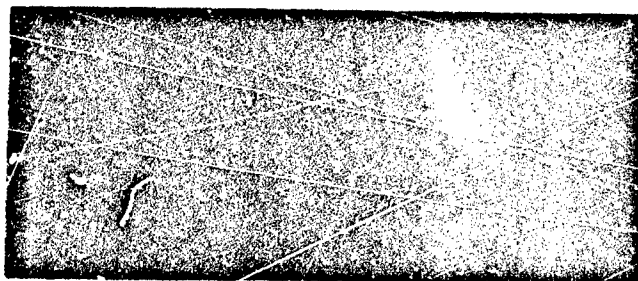   T=750°C
   thickness=4 μm

B) Sample #7176
   T=800°C
   thickness=5 μm

C) Sample #7177
   T=850°C
   thickness=8 μm

D) Sample #7178
   T=900°C
   thickness=15 μm

E) Sample #7179
   T=920°C
   thickness=15 μm

Figure 3.   Photomicrographs (1000X Magnification) Showing the
             Correlation Between Deposition Temperature (T) and
             Resulting Surface Morphology of InP Films on Glass
             Substrates.   (Marker represents 10 microns.)

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Annealing the as-deposited films at 850°C for 15 minutes yielded only a slight (barely perceptible under 1000X magnification) increase in grain size. The third grain enhancement technique, recrystallization, is described in Section 3.3.

3.3   Development of an InP Recrystallization Technology

The objective of this task was to enhance the grain size of the as-deposited film to dimensions exceeding 100 microns. It has been theoretically determined for polycrystalline InP films that this 100 micron minimum grain size is required to minimize electronic device losses at the grain boundaries [2]. In the first four months of this program, the as-deposited films had a grain size of 2-5 microns thus requiring substantial grain size enlargement to achieve the 100 micron goal.

AstroPower applied a zone melt recrystallization (ZMR) process to the as-deposited InP films as a grain enlargement process. Recrystallization is accomplished in the ZMR process by scanning a molten zone through the semiconductor film. The molten zone is created by a focused incoherent light [3]. The recrystallized film typically consists of large grains with a specific crystallographic orientation, and has electrical properties approaching those of bulk single crystalline material. Most investigators have found it necessary to use an encapsulant layer on top of the film to be recrystallized. The encapsulant serves to minimize contamination and decomposition during the ZMR process, and may induce a crystalline structure, improve surface morphology, and reduce or prevent agglomeration [4].

The main challenge in applying this technique was to develop an encapsulation layer to support orderly recrystallization, to inhibit the preferential escape of phosphorus and maintain the stoichiometry of the InP film.

The selection of possible encapsulants was made according to our previous experience with effective encapsulants for InP. However, our experience with InP has been limited to temperatures less than 700°C. The following encapsulants were used during the first three months of this program: silicon dioxide (SiO_2), yttrium stabilized zirconia (YSZ), and silicon nitride (Si_3N_4). The SiO_2 and YSZ films were 2000 Å thick and were deposited by electron-beam deposition. The Si_3N_4 films were 2000 Å thick and were deposited by chemical vapor deposition. Multilayer encapsulants were also used: Si_3N_4 (1600Å) over SiO_2 (2000Å) and Si_3N_4 (1600Å) over YSZ (2000Å).

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Under testing, all of the thin film encapsulants failed to fulfill the requirements dictated by the recrystallization process. Argon, oxygen, and nitrogen gas ambients were used. In all cases, the caps cracked allowing phosphorus to escape at temperatures below those required for recrystallization.

There are four possible failure mechanisms for the thin film encapsulants: i) poor adhesion of the cap to the InP film, ii) poor adhesion of the InP film to the glass substrate, iii) holes in the capping layer, and iv) inherent weakness in the containing capability of the encapsulant. The thin-film caps were applied to smooth and dense InP films. No visible adhesion problems (flaking or peeling) of the film/glass interface were evident. The caps were determined to be free of pinholes and cracks by visual inspection under 1000X magnification. It was concluded that the inherent containing strength of the thin-film caps was the limiting factor that caused the ZMR process to fail.

As an alternative to the thin-film caps, a mechanical cap was used. A diagram of the structure is shown in Figure 4. The purpose of this mechanical cap was to maintain the stoichiometry of the InP film by containing the evaporating phosphorus in close proximity to the as-deposited film.
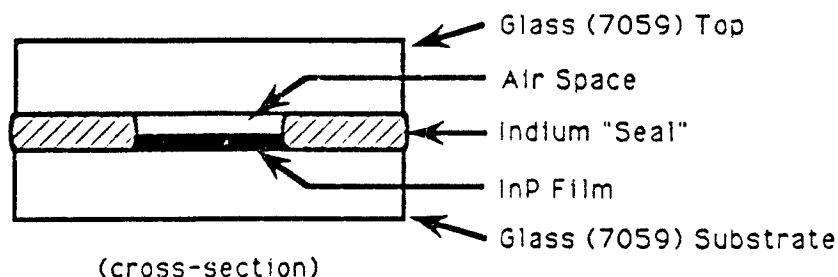

(cross-section)

Figure 4.   Mechanical Capping Structure (Case A).

The mechanical cap retained the constituents of the film within the air space of the structure, however the air space above the film was too large (about 1 mm in height) to suppress the complete dissociation of the film. Most of the film vaporized and was redeposited along the perimeter of the air space, along the indium seal. If the air space had been smaller (about 10 microns in height), phosphorus loss from the film would have been limited to the surface. The released phosphorus in the air space would then have been of sufficient pressure to suppress further (catastrophic) dissociation of the InP film. Controlling the air space height within micron dimensions was not possible with this structure, so additional experiments were completed with the air space completely filled with indium (case B).

During recrystallization of the mechanical capping structure that had no air space (case B), the film lifted off the glass substrate in one sheet and permanently attached itself to the

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bottom of the indium fill. This substrate/film separation was due
to the negative temperature gradient present along the cross-
section of the structure. The heat source was applied from the top
of the structure to keep the glass substrate as cool as possible.
(At substrate temperatures greater than 800°C the 7059 glass was
found to permanently warp.) The indium fill was hotter than the
glass substrate caus..ng the InP film to pull away from the cooler
glass substrate.

Note that in case B above, the substrate surface had been
sandblasted in a 1 cm^2 area prior to InP film deposition to
maximize adhesion. Areas of the film over both sandblasted and
non-sandblasted areas peeled off the substrate.

It was concluded that this mechanical capping structure would
not fulfill the requirements dictated by the recrystallization
process and would not be pursued further.

Because a successful encapsulant was not successfully
developed, development of the ZMR process for recrystallization of
InP films on insulating substrates was discontinued.
Characterization of films and fabrication of devices was completed
on as-deposited InP films on glass (i.e. no post-deposition grain
size enhancement techniques were used).

3.4   Characterization of the InP Films on Glass

Throughout this program, the material quality of the InP films
has been analyzed by optical (with Nomarski interference) and
scanning electron microscopy. Electron dispersive spectroscopy was
used to verify the stoichiometry of the InP films. No gross
impurities from the glass substrate were found in the films. The
device quality of the InP films was determined by fabrication and
analysis of Schottky diodes which will be discussed in detail in
Section 3.5.

The InP-on-insulator samples were subjected to device
fabrication processes: photolithography, chemical cleaning,
isolation etches. No change in appearance in the films was
observed; no cracking or peeling of the films occurred.

Hall measurements were performed to measure doping levels,
mobility, and conductivity type of the InP films. The conductivity
of the as-deposited film was found to be n-type; this was expected
since the source material is n-type (undoped) and no dopants are
introduced during the deposition process. The carrier
concentration of the films were measured to be 2×10^{16} cm^{-3}.
Accurate measurements of mobility could not be made due to
photolithographic complications with obtaining a thin enough strip
of material for accurate resistivity measurements.
Photolithographic masks specific to the requirements of these films

need to be designed and fabricated before accurate mobility measurements could be made.

3.5   Fabrication of Photodetectors from the InP Films on Glass

Photodetectors were fabricated on the five InP films shown in Figure 3 to demonstrate a correlation between the grain size and the device quality of the films. Several methods were used to fabricate the photodetector structures. These methods are described below.

3.5.1   Solid State Diffusion

The first photodetector design investigated was based on the formation of a p/n junction using solid state diffusion techniques. The as-deposited InP film would form the n-type base, and the p-type emitter would be formed by diffusing p-type impurities into that film. Two p-type dopants were used: cadmium and zinc.

The diffusion process was conducted in a liquid phase epitaxial boat and furnace. At diffusion temperatures (greater than 500°C), incongruent evaporation of phosphorus from InP surfaces results in severe pitting. Hence, the diffusion process had to incorporate protection against this thermal degradation. Two solutions were used for the diffusion process: 1) an InP overpressure melt with no dopant, and 2) an InP overpressure melt with dopant metal (zinc or cadmium). The InP overpressure melts are designed to prevent phosphorus loss from the InP surface without directly contacting the sample. The overpressure melts consist of indium (the solvent) and poly InP pieces (to provide the phosphorus overpressure); this combination provides a sufficiently high vapor pressure of phosphorus local to the InP sample surface to inhibit thermal degradation.

During the system transition from room temperature to the desired diffusion temperature, the sample was kept under an In/InP overpressure melt. Once the desired diffusion temperature was reached, the sample was moved to the diffusion melt. At the diffusion temperature the solid dopant source in the diffusion melt evaporates providing a partial pressure of the impurity in the gas over the sample. The impurity diffuses into the sample until the sample is removed from the vicinity of the diffusion melt and back to the first In/InP overpressure melt. The system is then quickly cooled to room temperature.

To determine the desired diffusion parameters (temperature, contact time), test samples of single-crystal InP LEC (Liquid-Encapsulated Czochralski) material were used. Cadmium was the first metal to be used as the dopant. No evidence of diffusion resulted using temperatures up to 600°C and contact times up to 30 minutes. Diffusion using elemental zinc as the dopant was

immediately successful; at 470°C with a contact time of 30 minutes, a 2.5 micron deep diffusion resulted uniformly across the sample.

However, repeating this zinc diffusion procedure on polycrystalline InP resulted in a diffusion depth that varied as much as 2 to 5 microns across the sample. This would prove detrimental to photodetectors that require a uniform emitter thickness of less than 0.1 micron to operate efficiently. This variation in diffusion depth is most likely due to a dependence of the diffusion coefficient on grain orientation (i.e. the zinc diffused faster in particular grains). This uniformity problem was also exacerbated by the rough surface texture of the InP films.

Due to these problems, solid state diffusion of a p/n junction for the InP films on glass was discontinued and Schottky barrier diodes were investigated.

3.5.2 Fabrication of Schottky Barrier Diodes

Figure 5 shows a cross-sectional diagram of the Schottky diodes fabricated during this program. Ohmic contact to the film bulk was made by electron-beam evaporation of a Au/Ge/Au/Ni contact along the perimeter of the sample. This contact was ohmic without alloying. The Schottky barrier was formed by the evaporation of two metals, 20 Å of zinc followed by 60 Å of gold, through a shadow mask. This shadow mask has an array of holes with diameters of 295 microns through which the Schottky metals were deposited.
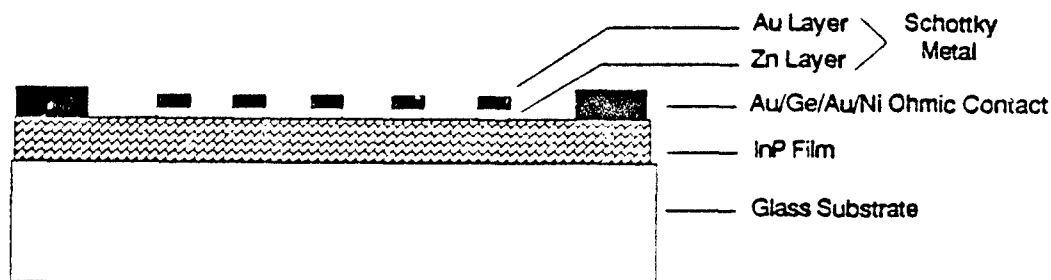

Figure 5. Cross-section Diagram of Schottky-Type Devices Fabricated from InP Films on Glass Substrates.

Seven samples were sent through an identical fabrication sequence: the five InP/Glass samples shown in Figure 3, a large grain commercial polycrystalline control sample, and a single crystal LEC control sample. Immediately after evaporation of the Schottky metal layers (zinc/gold), the devices showed ohmic characteristics, except for a control device on commercial polycrystalline material. The poly control sample showed a typical photosensitive diode characteristic. The Schottky metals on the remaining six samples were then annealed at 340°C for 2 minutes.

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The single crystal control and the largest grain InP film on glass (#7179, Figure 3E), showed photosensitive, diode characteristics. All other samples showed varying degrees of photoconductive response. Quantitative results are given in the next section.

3.5.3 Quantitative Device Performance

3.5.3.1 Photodiodes

Successful fabrication of Schottky-type photodiodes was demonstrated on three of the seven samples: the single crystal control, the polycrystalline control, and the largest grain InP/Glass sample (#7179, Figure 3E). The photogenerated current of these three devices is shown in Table 1.

TABLE 1
Measured Photogenerated Current Density
Under Incident Power of 135.3 mW/cm^2

Sample (Carrier Concentration)	Photogenerated Current (mA/cm^2)
Single Crystal Control (1×10^{17} /cm^3)	7.3
Polycrystalline Control (4×10^{15} /cm^3)	9.3
#7179 InP/Glass (2×10^{16} /cm^3)	10.7

About one hundred photodiodes were fabricated on each of the three samples. The current values given in Table 1 are average values taken from five representative devices on each sample. There were a few exceptional devices tested on the InP on glass sample that generated higher current densities under the same incident power. The best device on sample #7179 yielded 14 mA/cm^2. If a double antireflection coating were added to the device structure, this would correspond to a photogenerated current of 19.6 mA/cm^2. Note that this result is competitive with photogenerated current values that have been reported for photodetectors fabricated from single crystal InP material.

Table 1 provides the carrier concentration of the InP material for each sample. If photogenerated current were correlated with carrier concentration alone, regardless of material quality, it

would be expected that the magnitude of current would adhere to the following trend:

poly control > InP/Glass > single control   (1)

This would assume that material of lower carrier concentration yields longer diffusion lengths and more effectively collects the minority carriers that are generated. If, however, photogenerated current were correlated with grain size alone, regardless of carrier concentration, the following trend would be expected for the photogenerated current values:

single control > poly control > InP/Glass   (2)

The observed trend suggests that the smaller grain size of the InP film on glass does not limit the minority carrier collection of the device:

InP/Glass > poly control > single control   (3)

The dark I-V characteristic is a typical analytical tool for evaluating diode performance. The quality of the diode is established by the extraction of two important parameters: saturation current (the current value (I_o) measured as voltage approaches zero) and the ideality factor (n). Ideally, recombination and thermal generation of carriers occurs primarily in the neutral n-type bulk of the Schottky-type diode and n=1. Departure from a value of n=1 toward an n=2 indicates recombination within the depletion region of the device. The level of recombination is a measure of device quality; recombination centers arise from trace impurities or lattice defects which are known to accumulate at grain boundaries. Note that these same recombination centers contribute a carrier generation source within the depletion region thus increasing the saturation current value above the theoretical value that would be expected from perfect quality material. The dark I-V characteristic for a typical Schottky diode fabricated on the single crystal control, polycrystalline control, and #7179 InP/Glass are shown in Figure 6. Approximations of the saturation current (I_o) for each sample are also given.
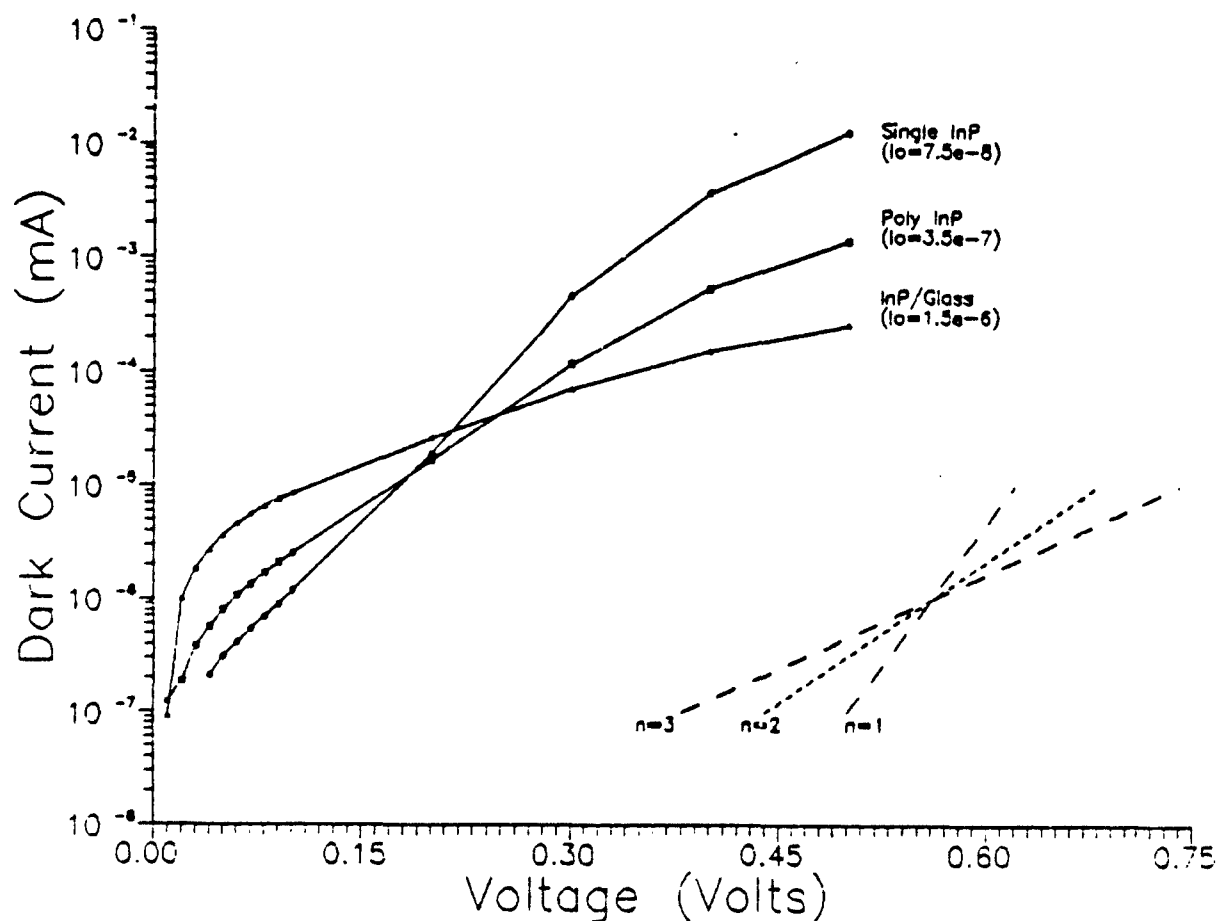
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Figure 6. Photodetector Dark I-V Characteristic.

Although Schottky diodes are very easily fabricated and provide informative routes for material analysis, their performance is typically limited by the presence of an extra parasitic current component in addition to those associated with p-n junctions [5]. This extra parasitic component acts to increase the dark saturation current of the diode. This explains, in part, the relatively high saturation current density of all of the devices shown in Figure 6 compared to the theoretical value (2.8 x 10^{-19} mA for the same size device as those in Figure 6) expected from InP's bandgap of 1.35 eV.

Figure 6 shows the dark characteristic line slopes (dotted lines) expected from diodes with ideality factors of n=1, 2, or 3.

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The single crystal control approximates an n=1, the polycrystalline control approximates an n=2, and the InP/Glass characteristic suggests an n>2. Although no widely accepted explanation for a diode with an ideality factor of n>2 exists, several contributing loss factors may be held accountable: i) recombination centers in the depletion region, ii) recombination centers at grain boundaries, and iii) obscuration of dark I-V curve by series resistance and shunt resistance effects. The I-V characteristic for InP/Glass detectors under illumination (not shown here) was dominated by series resistance and shunt resistance effects. These account for the decreasing slope (series effect) of the dark I-V curve as voltage increases and the increasing slope (shunt effect) as voltage approaches zero.

Unlike the control samples, the performance of the devices on the InP on Glass sample varied across the film. This is not surprising. The grain size of the film was approximately 10 microns, and the device diameter was approximately 295 microns. Hence, each device incorporated numerous and varying crystal orientations and grain boundaries. Grain boundaries are known to strongly influence the electrical characteristics of thin films. Three aspects of grain boundary phenomena that have been identified as being important for photovoltaic operation in polycrystalline semiconductor materials [6] were also observed during this program:

1. The potential energy barriers associated with grain boundaries dominate the carrier mobility and control resistivity resulting in series resistance losses.

2. The defect and impurity states that occur at the grain boundaries cause recombination of excess carriers that ultimately reduce the effective minority carrier lifetime in an inhomogeneous manner.

3. The penetration of the device depletion layer by the disordered regions of the grain boundary interiors can lead to shunting conductance and increased diode current.

The use of a conducting coating (e.g. indium tin oxide, ITO) over the thin film would ease carrier transport parallel to the film so that the effects of polycrystallinity would not strongly effect resistivity and series resistance loss could be minimized. The solution to shunting conductance, increased diode current, and increased recombination, is to increase the grain size of the films.

3.5.3.2 Photoconductors

The four remaining InP on Glass samples did not yield rectifying (diode) characteristics. Although these samples underwent the same device fabrication process as the three samples described above, the resulting devices responded as photoconductors

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rather than as true photodiodes. The observed trend in device performance suggests that the diode characteristic softens with decreasing grain size, until it can no longer be called a photodiode and is showing only basic photoconductive behavior. It was concluded that the grain sizes of the InP films of samples #7178, #7177, #7176, and #7171 were too small relative to the device area to adequately support photodiode behavior.

Table 2 lists the photogenerated current of the photoconductors tested on the smaller grain InP films on glass. Because device performance varied across the film, and there were over one hundred devices per sample, both the lowest and highest photogenerated currents measured for the particular sample are given. Note that the magnitude of the photogenerated current correlates with the grain size of the InP film (as shown in the photomicrographs of Figure 3). As the grain size approaches the mean optical absorption length $1/\alpha$ (depends on the wavelength of light, approximately 0.8 micron at the band edge), the number of photogenerated carriers lost to recombination at the grain boundaries increases.

TABLE 2
InP/Glass Photogenerated Current
Under Incident Power of 135.3 mW/cm²

Sample	Photogenerated Current (mA/cm²)	
	LOW	HIGH
#7171	0.01	0.20
#7176	0.44	0.70
#7177	0.30	0.60
#7178	7.40	16.20

4.0 SUMMARY OF RESULTS

This program demonstrated the growth of InP thin films on glass of sufficient quality to support minority carrier devices. A low cost deposition technique was established which produced mechanically stable, stoichiometric InP films on insulating substrates. Device quality was established by the fabrication and successful operation of Schottky barrier diodes. Device performance comparisons of these devices with identical structures on large grain commercial polycrystalline InP material and LEC single crystal InP material provided a relative basis for analysis of material quality. Device performance was dependent on and

limited by grain size. Larger grain material (greater than 10 micron dimensions) is required for further analysis and optimization of the InP films on glass.

At the beginning of this program, it was expected that zone melt recrystallization would be used to enhance the grain size of the as-deposited InP films to 100 μm. However, an encapsulant that could contain the film's constituents during recrystallization was not found. As a result, the device results reported here are based on InP films with as-deposited morphologies offering up to 10 micron grains.

5.0 ESTIMATES OF TECHNICAL FEASIBILITY

The quality of the InP/Glass photodiodes fabricated and tested during this program indicate the high device potential of these films for grain sizes exceeding 10 microns. If an adequate encapsulant could be found to contain the as-deposited InP film and maintain its stoichiometry during recrystallization, then further optimization of the InP films on glass could be achieved. Larger grain InP films are needed to provide more conclusive findings on the optimum grain size for device applications.

6.0 REFERENCES

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